

THE INSTITUTE OF PAPER CHEMISTRY

Appleton, Wisconsin

DEVELOPMENT OF A MANUFACTURING PROCEDURE FOR LOW-LITHIUM,
LOW-URANIUM CONTENT FILTER PAPER

Project 3101

Report Eleven

A Status Report

to

DEPARTMENT OF THE AIR FORCE
1155th TECHNICAL OPERATIONS SQUADRON (HQ. COMD.)
McCLELLAN AFB, CALIFORNIA

March 5, 1974

TABLE OF CONTENTS

	Page
SUMMARY	1
INTRODUCTION	2
DISCUSSION	10
FUTURE WORK	11
EXPERIMENTAL	12
LITERATURE CITED	12

THE INSTITUTE OF PAPER CHEMISTRY

Appleton, Wisconsin

DEVELOPMENT OF A MANUFACTURING PROCEDURE FOR LOW-LITHIUM,
LOW-URANIUM CONTENT FILTER PAPER

SUMMARY

Additional data were obtained for the uranium and lithium contents of simulated handsheets and for purified lots of IPC-1478 paper, Series N. Handsheets made from purified pulp and commercially-made paper purified by a percolation technique contained < 0.1 ng U/g and 0.7-1.5 ng Li/g. Work will continue to reduce the lithium content to the specified goal of < 0.4 ng Li/g.

Tables I-IV in this report combine the new data with those previously reported in Project 3101, Report Ten, Tables I-IV.

INTRODUCTION

Tables I-IV are duplicates of the tables in Report Ten, January 22, 1974, except that additional data, supplied by McClellan AFB, MCL-C, have been added. Report Ten should be consulted for experimental details.

TABLE I

SIMULATED HANDSHEETS MADE IN ALL-PLASTIC EQUIPMENT^a

Sample No.	Sheet No.	Uranium		Lithium,	
3101-		8/5	ng/g	ng/g	
<u>Handsheet Series 231-240.</u> Purified pulp (3101-193 to 195, Report Eight, September 13, 1973) was made into 50-g handsheets in an all-plastic apparatus (except stainless steel stirrer). The white water (filtrate) from each hand-sheet was recycled; total volume of water was 5 liters for 425 g. of handsheets.					
231	1 (50 g)	A	137	0.32	1.66
		B			2.48
232	2 (25 g)	A	134	0.19	3.30
		B			1.05
		C	134	0.17	0.95
237	9,10 (50 g, ea.)	A	135	0.14	1.35
		B			0.70
240	15,16 (50 g, ea.)	A	137	[0.89] ^c	1.34
		B	135	0.19	1.47
241	White water (5 l) from 231-240			0.017 ng/ml	0.059 ng/ml

Handsheet Series 242-247. The general procedure as described above for handsheet Series 231-240 was used except that 10-g sheets were formed; total volume of water was 3 liters for 145 g of handsheets.

242	1 (20 g)	A			-- ^b
		B	134	0.13	2.22
		C	139	0.13	1.01
243	2 (15 g)	A			-- ^b
		B			1.42
		C	139	0.11	1.01
245	5,6,7 (10 g, ea.)	A	133	0.14	-- ^b
		B			0.96
		C	136	0.09	0.86
247	11,12,13 (10 g, ea.)	A	133	0.13	-- ^b
		B			1.57
		C	134	0.13	1.15
248	White water (3 l) from 242-247		137	0.006 ng/ml	0.041 ng/ml

See end of table for footnotes.

TABLE I (Continued)

SIMULATED HANDSHEETS MADE IN ALL-PLASTIC EQUIPMENT^a

Sample No.	Sheet No.	Uranium		Lithium,
3101-		8/5	ng/g	ng/g
<u>Handsheet Series 249-264.</u> The general procedure as described above for hand-sheet Series 231-240 was used except that a small Buchner funnel was substituted for the large table top Buchner when the latter became clogged with fines; total volume of water was 3.7 liters of water for 565 g of handsheets.				
249	1	A	133	0.18
		B		-- ^b
		C		1.66
				1.06
250	2	A	129	0.18
		B		1.02
				1.01
251	3	A		-- ^b
		B	136	0.09
		C		0.77
				0.62
(small Buchner substituted for large Buchner)				
254	8,9,10	A	137	0.16
		B		-- ^b
		C		0.63
		D	134	0.09
				1.92
257	17-20	B	136	0.10
				1.01
260	31-35		129	0.11
				2.41
264	51-53	A	131	0.22
		B		2.21
				2.88
265	White water (1.7 l, final volume) from 249-264		132	0.009 ng/ml
				0.13 ng/ml

^aSee letter dated October 17, 1973, E. E. Dickey to Captain Grosso for a complete list of samples and letter dated November 29, 1973, Captain Grosso to E. E. Dickey for confirmation of data in this table.

^bResults discarded.

^cResult appears to be in error.

TABLE II

LEACHING OF HERCULES PULP (PS-57), IPC-1478 PAPER
 (SERIES N), AND COTTON SCRIM^{a,e}

Sample No.			Uranium	Lithium,	
3101-		8/5	ng/g	ng/g	Remarks
3.6 Kg Hercules PS-57 pulp. Leached with 1.5 liters of water; 3 liters of 0.1M ammonium carbonate; 10 liters of water; 4 liters of 0.1M hydrofluoric acid; 12 liters of water; dried; cycle repeated once.					
266	A			0.22	
	B	135	0.04	0.10	
2.2 Kg Hercules PS-57 pulp. Leached with 1.0 liter of water; 4 liters of a solution 0.1M in both ammonium carbonate and ammonium fluoride; 7.7 liters of water; dried; cycle repeated once.					
First cycle					
267	A	117	0.29	0.40	
	B			0.35	
Second cycle					
268	A			0.63	
	B	118	0.15	0.34	
Stack of Hercules pulp PS-57 (516 g) in squares (15.5 cm) with scrim at one-third and two-thirds the distance down the stack. Leached with 2 liters of 0.1M ammonium carbonate; 2 liters of water; 2 liters 0.1M hydrofluoric acid; 3.5 liters of water; dried; cycle repeated once.					
271	A	139	0.19	3.34	Scrim, No. 9994 ^c , 16 g
	B			3.30	
MCL No.					
013-5039H (control)				68.1	Scrim, No. 9994 as received ^c
273	A	133	0.05	0.24	Pulp, Hercules PS-57 from zone intermediate between scrim Samples 271 and 275
	B			0.25	
275	A	137	0.16	3.05	Scrim, No. 9995 ^c , 18 g
	B			3.74	
MCL No.					
013-5039I (control)				80.0	Scrim, No. 9995 as received ^c

See end of table for footnotes.

TABLE II (Continued)

LEACHING OF HERCULES PULP (PS-57), IPC-1478 PAPER
(SERIES N), AND COTTON SCRIM^{a,e}

Sample No.			<u>Uranium</u>	Lithium,	
3101-			8/5	ng/g	Remarks
Stack (total weight, 747 g) of Hercules pulp, PS-57 and IPC-1478 paper in 17 cm squares; leached with 2 liters of 0.1M ammonium carbonate; 2 liters of water; 2 liters of 0.1M hydrofluoric acid; 5 liters of water.					
278	A	125	0.11	1.82	First cycle. IPC-1478 paper (see No. 294) Scrim only, removed from IPC-1478 paper
	B			1.46	
	C	131	0.24	4.89	
280	A	133	0.06	0.99	Second cycle. IPC-1478 paper from middle section (see No. 294) Scrim only, removed from IPC-1478 paper
	B			1.07	
	C	137	0.13	3.93	
282	A	131	0.03	0.56	Pulp after second cycle, middle section of stack
	B			0.49	
Stack (total weight, 515 g) of Hercules pulp (PS-57), IPC-1478 paper, and cotton scrim was leached with 2 liters of 0.1M oxalic acid; 5 liters of water.					
288	A	127	0.15	1.84	IPC-1478 paper ^b
	B			1.53	
289	A			7.37	Scrim ^{c,d}
	B	129	0.39	7.45	
290	A	120	0.11	0.39	Pulp squares
	B			0.56	
Stack (similar to 288-290) of Hercules pulp (PS-57), IPC-1478 paper, and cotton scrim was leached with 2 liters of 0.02M EDTA (disodium ethylenediaminetetraacetate); 5 liters of water.					
291	A	121	1.02	2.80	IPC-1478 ^b Scrim removed from IPC-1478 paper
	B			2.31	
	C	129	1.16	8.11	
292	A			8.56	Scrim ^{c,d}
	B			8.34	
293	A			0.51	Pulp squares
	B			0.23	

See end of table for footnotes.

TABLE II (Continued)

LEACHING OF HERCULES PULP (PS-57), IPC-1478 PAPER
(SERIES N), AND COTTON SCRIM^{a,e}

Sample No.	Uranium	Lithium,	Remarks
3101-	8/5 ng/g	ng/g	
294 (control)			
A		24.7	
B		24.0	IPC-1478 paper, Series N

^aSee letter dated October 17, 1973, E. E. Dickey to Captain Grosso for a complete list of samples.

^bThe IPC-1478 paper, untreated with Kronisol, was cut from Roll No. 22, Series N.

^cSee letter dated September 24, 1973, Captain Grosso to E. E. Dickey.

^dThe scrim was cut from Roll G, No. 9993, with a lithium content of 32.8 ng Li/g.

^eData confirmed and expanded in letters dated November 29, 1973 and February 4, 1974, Captain Grosso to E. E. Dickey.

TABLE III

LITHIUM CONTENT OF PURIFIED IPC-1478 PAPER CONTAINING KRONISOL^c

Sample No.	Weight, g	Uranium 8/5 ng/g		Lithium, ng/g		Description
<u>Purification of IPC-1478 Paper (Series N) and Addition of Kronisol.</u> An amount of 92 g (23 squares) of the paper were placed in a block between pulp squares, 137 g above and 81 g below the paper. The stack was leached by percolation in two cycles with 2.5 liters of 0.1M ammonium carbonate, 2.5 liters of water, and 2.5 liters of 0.1M hydrofluoric acid, 3.0 liters of water, and dried. Kronisol, 27 g, was distributed through the block of paper squares by capillary migration and centrifugation.						
308 ^a	133					Pulp squares, top section, from Sample 309
309	87	A	131	0.03	0.68	IPC-1478 paper, Series N, with Kronisol, top 36.8%, middle 37.2%, bottom 8.7% of block of paper ^b
		B	132	0.05	0.95	
		C			0.76	
		D	133	0.07	1.54	
		E	135	0.06	1.48	Scrim removed from A-D, combined
		F	132	0.08	1.08	
		G	127	0.07	0.88	
		H	127	0.06	0.95	
		I			1.27	
		J	120	0.10	1.95	
		K	136	0.11	1.37	Scrim removed from F-I, combined
		L	134	0.11	1.11	
310 ^a	79					Pulp squares, bottom section, from Sample 309
"Old" series IPC-1478 paper						Kronisol content, 13.6%

^aThese samples are retained at the Institute and are available on request.

^bSix squares of paper, two each from the top, middle, and bottom of the block were removed for Kronisol analysis by direct extraction with acetone.

^cThese results, in part, were transmitted by telephone on December 17, 1973, from Captain Grosso to E. E. Dickey and were confirmed and extended in a letter dated February 4, 1974, from Captain Grosso to E. E. Dickey.

TABLE IV

LITHIUM CONTENT OF FINES COLLECTED FROM HERCULES PULP, PS-57^b

Sample No. 3101-	Weight, g	Uranium		Lithium, ng/g ^b	Description
		8/5	ng/g		
295 (control)	120	A	116	1.14	Hercules pulp, PS-57
		B		2.11	
<u>Collection of Pulp Fines.</u> Pulp (Hercules PS-57), 15 g, was dispersed in 1.2 liters of water; slurry filtered on circle of pulp; filtrate recycled for successive 15 g lots of pulp; total amount of pulp, 345 g; total volume of filtrate 2.8 liters.					
296 ^a	--				Circle No. 1. Fines from Sample No. 297 (Pads 1, 2, 3)
297 ^a	45				Pads 1, 2, 3
298	--	A		2.66	Circles No. 2, 3. Fines from
		B		2.75	Samples 299 and 300 (Pads 4-11)
299	60	A		1.25	Pads 5, 7, 9, 11
		B	116	1.14	1.32
300 ^a	60				Pads 4, 6, 8, 10
301 ^a	--				Circle No. 4. Fines from Sample 302 (Pads 12, 13, 14, 15)
302 ^a	60				Pads 12, 13, 14, 15
303	--	A		2.80	Circles No. 5, 6. Fines from
		B	116	2.11	Samples 304 and 305 (Pads 16-23)
304	60	A		1.28	Pads 17, 19, 21, 23
		B		1.32	
305 ^a	60				Pads 16, 18, 20, 22
306 (control)	--	A		2.21	Circles of Hercules PS-57
		B	116	1.27	2.45
307	--	A		0.285 ng/ml	White water (2.8 liters)
		B	116	0.0088 ng/ml	from Samples 296-305

^aThese samples are retained at the Institute and are available on request.

^bThese results, in part, were transmitted by telephone on December 17, 1973, from Captain Grosso to E. E. Dickey and were confirmed and extended in a letter dated February 4, 1974, from Captain Grosso to E. E. Dickey.

DISCUSSION

New data combined with those previously reported reinforce the previous observations and conclusions about the production of low-lithium paper (1,2). The data for simulated handsheets (Samples 3101-231 to 265, Table I) made from purified pulp support the conclusion that IPC-1478 paper containing low levels of uranium and lithium may be produced in special equipment. Possibly plastic and stainless steel equipment located in a dust-free room and supplied with purified water would be essential.

The data in Tables II and III support the conclusion that IPC-1478 paper, made commercially, may be processed subsequently to contain low levels of uranium and lithium. The method appears to be practical and capable of being scaled-up to meet limited demands for such paper. The equipment and environment would be similar to those of a process for the purification of pulp. Thus, the purification of IPC-1478 paper, Series N, with the application of Kronisol resulted in very low levels of uranium (generally < 0.1 ng U/g) and lithium contents of 0.7-1.5 ng Li/g (Table III).

In a limited attempt to determine the relationships of fines to the uranium and lithium contents of IPC-1478 paper, the data (Table IV) show that the fines may be significantly higher in lithium than the original pulp (compare Samples 298 and 303 with 299, 304, and the controls). The uranium content of Sample 303 was somewhat higher than that of 299 and the controls, a behavior similar to that of lithium.

FUTURE WORK

1. Five series of handsheets were made in an all-plastic sheet mold. The analysis of these samples is in process at McClellan AFB, MCL-C.
2. Samples of IPC-1478 paper, Series N, have been treated with ammonium bicarbonate, ammonium fluoride, and ammonium chloride. The analysis of these samples is in process.
3. Two lots of IPC-1478 paper, Series N, were treated with ammonium carbonate and hydrofluoric acid, and then with Kronisol. These samples will be shipped on or before March 1, 1974 to McClellan AFB for analysis.
4. An attempt may be made to purify squares (20 inches x 20 inches) of IPC-1478 paper and to add Kronisol.


EXPERIMENTAL

The experimental details for the samples listed in Tables I-IV were reported in Report Ten, January 22, 1974.


LITERATURE CITED

1. Project 3101, Report Nine, November 9, 1973.
2. Project 3101, Report Ten, January 22, 1974.

THE INSTITUTE OF PAPER CHEMISTRY



Edgar E. Dickey
Senior Research Associate
Division of Natural
Materials & Systems



Irwin A. Pearl
Group Coordinator
Division of Natural
Materials & Systems